



Contribution ID: 70

Type: **Oral contribution**

Development of liquid and electrochemical cells for operando NAP-XPS/NEXAFS investigations

Tuesday, 6 December 2022 16:00 (20 minutes)

To track the chemical state and structural properties of catalytically active materials under realistic reaction conditions, in order to understand charge transfer at the liquid-solid interface, CO₂ activation, and surface carbon intermediate species, reaction cells are critical for operando near ambient soft X-ray photoelectron and absorption spectroscopy (NAP-XPS/XAS) experiments. The main challenges with developing operando NAP-XPS/XAS cells for photocatalytic and electrocatalytic reactions are that these reactions are typically performed in liquid phase at the solid-liquid interface and that the catalyst must be exposed to UV-visible irradiation during reaction (and apply an external potential in both electrocatalysis and photoelectrocatalysis). The presence of the liquid phase alone poses a challenge due to the high absorbing background, but external excitation with UV-visible light or electrons imposes additional limitations on cell design. In order to address these challenges while probing catalyst surface during the reaction, we have developed back-side illuminated operando reaction cells for synchrotron-based NP-XPS AND NEXAFS studies. Our design concept is based on a modular design and uses non-metal (PEEK material) body, and replaceable membranes which can be either of X-ray transparent silicon nitride or of water permeable polymer membrane materials (e.g. Nafion). These membrane materials are particularly suitable for liquid flow or electrochemical cells and enable measuring photoelectrons emitted from the membranes or from catalyst material deposited at the solid-liquid interface outside the cell. The preliminary results of in-situ NAP-XPS and NEXAFS measurements (from B07 beamline@Diamond) on electrochemical reduction with a Cu catalyst and oxidation with an IrO_x catalyst will be presented. The developed system is highly modular and can be used in the laboratory or directly at the beamline for operando XPS/XAS measurements (Figure 1).

if "Other", please specify:

I apply for a travel grant

No

Primary author: KUMAR, Santosh (Diamond Light Source)

Co-authors: Dr GRINTER, Dave (Beamline Scientist); Prof. HELD, Goerg (Principal Beamline Scientist); Mr COUNTER, James (PhD student); Dr V. SPRONSEN, Matthijs (Beamline Scientist); Dr FERRER ESCORIHUELA, Pilar (Beamline Scientist)

Presenter: KUMAR, Santosh (Diamond Light Source)

Track Classification: Other - please specify below